Small Angle X-ray Scattering Studies of Argon in Xerogel

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Introduction: Confinement in disordered porous material has been known to affect the thermodynamic properties of imbibed liquid and solid phases. Confinement can not only change the microscopic structure of the adsorbed solid, but also eliminate or introduce new transitions or stabilize new phases. For example, studies of Ar and Kr in vycor glass report crystallization structures different from those in the bulk.

Methods and Materials: The Ar was confined in both powdered xerogel (Britesorb®) and powdered vycor glasses. The vycor and xerogel have nominal pore diameters of 70Å and 130Å and porosities of 30% and 72%, respectively. The powders were tightly packed into a cylindrical cell, with Be windows to allow the passage of x-rays. The cell was mounted on a closed cycle refrigerator and the temperature monitored with a silicon diode thermometer. The samples were filled with Ar such that they were 95% full at zero vapor pressure.

Results: In the previous abstract, **X-ray Diffraction Studies of Ar in Confined Geometries**, we showed evidence of a mobility transition at a temperature much lower than the freezing temperature (please see this abstract for information on sample properties and cell geometry). To further explore the nature of this transition, Small Angle X-ray Scattering (SAXS) was employed. SAXS can give information on properties such as the pore size of the matrix. More importantly for our measurements, SAXS gives information on the macroscopic distribution of the scatterers. This can allow us to see changes in the structure of the Ar, i.e. liquid-solid transition or solid-solid transition. If the Ar is leaving the pores, one should be able to see changes in the scattering at these very low *Q*'s.

Fig. 1 shows SAXS measurements of powdered xerogel filled with Ar at various temperatures as well as an empty cell scan. After filling the pores, the scattering decreases and changes shape. The change in shape reflects the fact that the primary scattering is now Ar and no longer the xerogel matrix. The liquid (85K) and two high temperature solid measurements are quite similar in shape, although the intensity has decreased slightly in the solid. It is important to note that the 60K and 70K solid measurements are identical in both shape and intensity. Neither time nor temperature plays a part in the high temperature solid phase. Fig. 2 shows SAXS measurements at temperatures of 60K as well as 48K at three different times. In the lower temperature the intensity increases as a function of time and appears to follow a linear dependence.

Conclusions: Clearly, there has been a significant change in the structure of the Ar giving more credence to the mobility transition discussed in our previous abstract.

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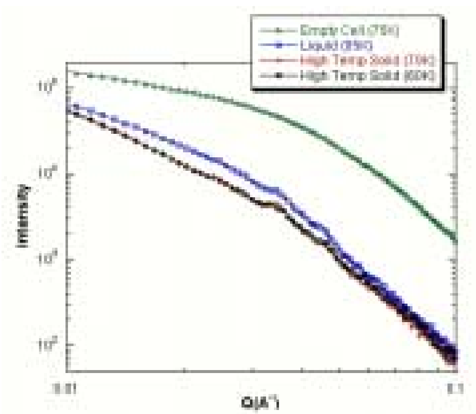

Figure 1. SAXS measurements for the empty cell, liquid phase, and two solid phases. Note that the two solid phases are identical.

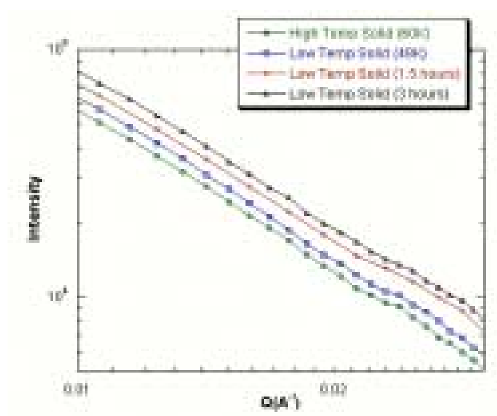

Figure 2. SAXS measurements for a solid phases at 60K and 48K. Note the increase in intensity as a function of time for the low temperature solid phase